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13. ABSTRACT

Dry ashing and extn. of a grease soln. in C_6H_6 with HCl were used to det. Zn in greases for protecting galvanized cables. The further complexometric detn. of An was less sensitive than dithizone titrn. and polarog. detn., which took 20 to 25 min. [AP1203743]

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Polarographic Analysis						
Grease						
Chemical Composition						
Protective Coating						
Zinc						

U. S. BOARD ON GEOGRAPHIC NAMES TRANSLITERATION SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	*А а*	A, a	Р р	*Р р*	R, r
Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

* ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ё. The use of diacritical marks is preferred, but such marks may be omitted when expediency dictates.

DETERMINATION OF THE ZINC CONTENT IN HYDROCARBON LUBRICANTS

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The production of hauling, road-construction and other equipment having a high degree of reliability, in which the most important and integral parts are the galvanized cables, involves the development of new lubricating materials having raised requirements for protective purposes. The protective properties of the lubricating materials can be evaluated according to the amount of build-up of the corrosive products (ions of the metal) in the lubricants over a determined period of time. There are a number of methods (X-ray spectral [1, 2], atomic-absorption [3] and activation) for quantitative determination of the zinc content in lubricants.

The use of the spectral method [4-6] involves a number of difficulties, in particular, with the preparation of the sample. The most suitable methods in this case are those associated with the transfer of the zinc from the analyzed sample to the aqueous solution [6-9][1].

[1]The most complete listing of works in which the various methods of determining metals in petroleum products are described can be found in reviews published in the journal Analytical Chemistry. For example, Analyt. Chem., 1969, 41, No. 5, 169.

For the determination of zinc in an aqueous solution it is expedient to use the complexometric, extraction-dithizone or polarographic methods [10].

For the transfer of metals from petroleum products to an aqueous solution, extraction [8-9] as well as dry and moist ashing are widely used. In [11] ion exchange in a nonaqueous medium is used for the transfer of copper from transformer oil to an aqueous solution.

In this work, dry ashing and extraction were used for the transfer of zinc from the lubricant to the aqueous solution. In the aqueous solution zinc was determined by complexometric titration. extraction titration with a solution of dithizone in carbon tetrachloride ~~[12]~~, and polarographically.

The polarographs were recorded on an ON-101 polarograph in a conical air-tight chamber. An ammonia buffer (3N NH_4Cl + 0.4N NH_4OH) containing 10^{-3}% gelatin [13] was used as background.

The titers of the solutions of dithizone in carbon tetrachloride (extraction titration) and Trilon B in water (complexometric titration) were established on the basis of a solution of zinc prepared from a sample of metallic zinc, brand [TsO] (ЦО).

For the transfer of zinc into an aqueous solution two methods of extraction were used. According to the first method the zinc was extracted from a benzene solution of lubricant per [GOST] (ГОСТ) 13538-68 (double extraction with hydrochloric acid, 25 ml each) using 6N hydrochloric acid, The hydrochloric acid solution of zinc chloride was boiled down to a volume of 5-7 ml for the removal of n-butyl alcohol and water. The remaining solution was transferred to a graduated 100 ml flask, and the residues were washed from the walls of the beaker with hot doubly-distilled water in the same flask. Zinc was determined from the obtained solution using the

three above-mentioned methods (Table 1).

Table 1 The content of zinc in Torsiol-55 lubricant ($1 \cdot 10^{-2}$ wt%) after testing in a special corrosive medium (extraction according to the first method).

Trilonometric titration	Dithizone titration	Polarographic determination
4.60	4.40	4.37
4.80	4.60	4.45
6.74	6.20	6.13
6.35	6.00	5.73
7.42	7.43	7.44

The second method of extraction (single extraction of a lubricant dissolved in a sufficiently small quantity of benzene, 6N aqueous solution of hydrochloric acid) by one of the authors of this paper was used earlier [14] during the polarographic determination for zinc using additives of the zinc dialkyl dithiophosphate type. Extraction was done in a separating funnel at room temperature. This method was used for the polarographic determination of zinc in lubricants.

The method of dry ashing is the evaporation of a lubricant in a crucible over a gas burner until there is a dry residue, and its calcination in a muffle furnace (at a temperature of 750°C) for 2-2.5 hr. The obtained ash is treated with 5 ml of concentrated hydrochloric acid during heating under a watch glass, then boiled down to almost one-half; the contents of the crucible are transferred to a graduated 100 ml flask, with the residue carefully washed from the walls of the crucible and from the watch glass using hot doubly-distilled water.

The use of dry ashing and extraction by the first method gives results that agree quite well (Table 2). In connection with this, since the ashing of the lubricant proceeds very slowly (over several days), only the extraction methods are used for the transfer of zinc from the lubricant to the aqueous solution.

Table 2. Content of zinc ($1 \cdot 10^{-2}$ wt%) in lubricants after testing in a IP-1-3 Chamber (complexometric method).

Lubricant	Extraction according to the first method	Ashing
Commercial vaseline	0.71	0.56
	1.06	1.03
	1.20	1.20
Torsiol-55	0.79	0.60
	0.88	0.86
	1.74	1.90

The accuracy of the methods of zinc determination were checked by analyzing samples of the lubricants with known quantities of zinc. These samples were treated with additions of Lubrizol containing 8.79% zinc (determined according to GOST 13538-68) and with additions of Torsiol-55 containing no zinc. In order to attain homogeneity of the mixture, the lubricants and additives were melted together and thoroughly mixed. The mixing is repeated after cooling and prior to each sampling. The analytical data of two such artificially treated mixtures (Table 3) indicate that all the employed methods give sufficiently accurate and agreeing results.

Table 3. Content of zinc ($1 \cdot 10^{-2}$ wt%) in artificial mixtures (Torsiol-55 lubricant, Lubrizol additive).

Calculated content of zinc	Extraction		
	By the first method		By the second method
	Trilonometric titration	Extraction, titration with dithizone	Polarographic determination
2.08	1.99	2.10	2.10
	2.04 (0.03)*	2.20 (0.06)*	2.00 (0.06)*
	2.04	2.20	2.00
0.28	---	0.29	0.25
	---	0.28	0.29 (0.03)*
	---	---	0.29

*Absolute rms error of one measurement.

The sensitivity of the complexometric method is not as good as that of the dithizone and polarographic methods. It cannot be used for the determination of less than $0.5 \cdot 10^{-2}$ wt% of zinc. The dithizone and polarographic methods in this respect are approximately equal in feasibility. The dithizone method does not require special equipment; however, the frequent adjustment of the titer of the dithizone solution (the dithizone solution is unstable [12]) and the preliminary preparation of the sample for analysis are time-consuming. Therefore, with the necessary equipment it is more expedient to use the polarographic method for the determination (extraction by the second method) in which the analysis of a single sample can be completed in 20-25 min. Timewise this is significantly more economical in comparison with other methods.

All three of the above-described methods have been successfully used for analysis over a long period of time at the All-Union Scientific Research and Planning Institute for Underground Gasification of Fuels--Petrochemical Branch, and they all provide good corresponding results. They are recommended for more extensive application.

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